STRESS CORROSION TESTING OF FULL-THICKNESS DIGESTER WELDMENTS

A Report to the TAPPI/IPC Digester Cracking Research Committee

by

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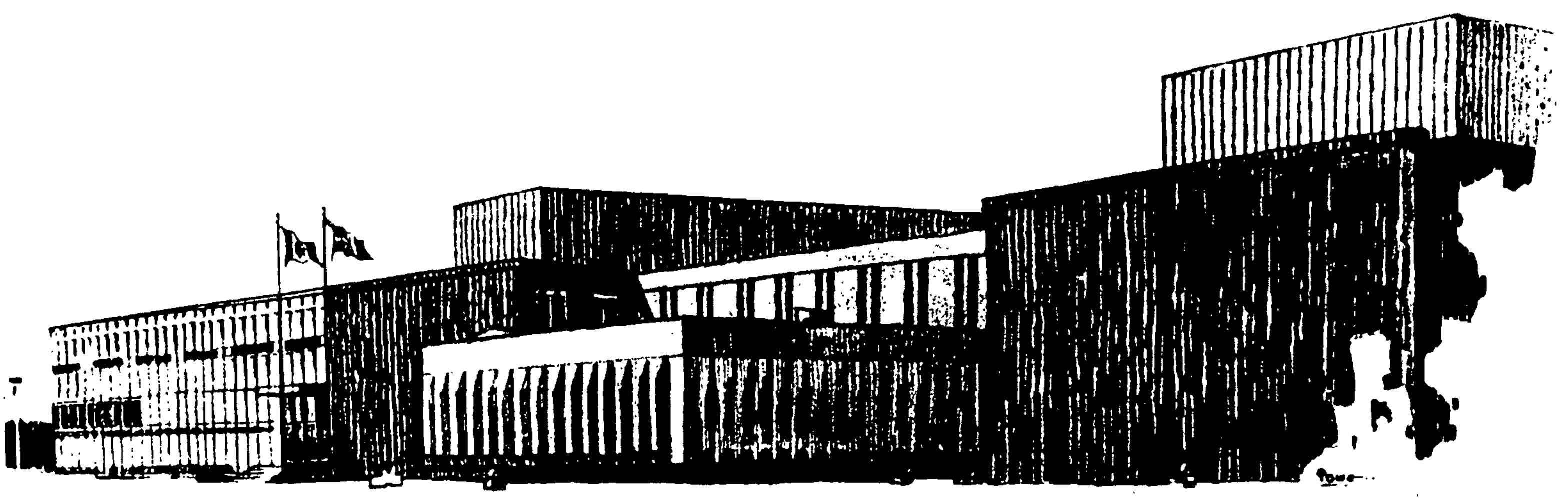
September 8, 1986



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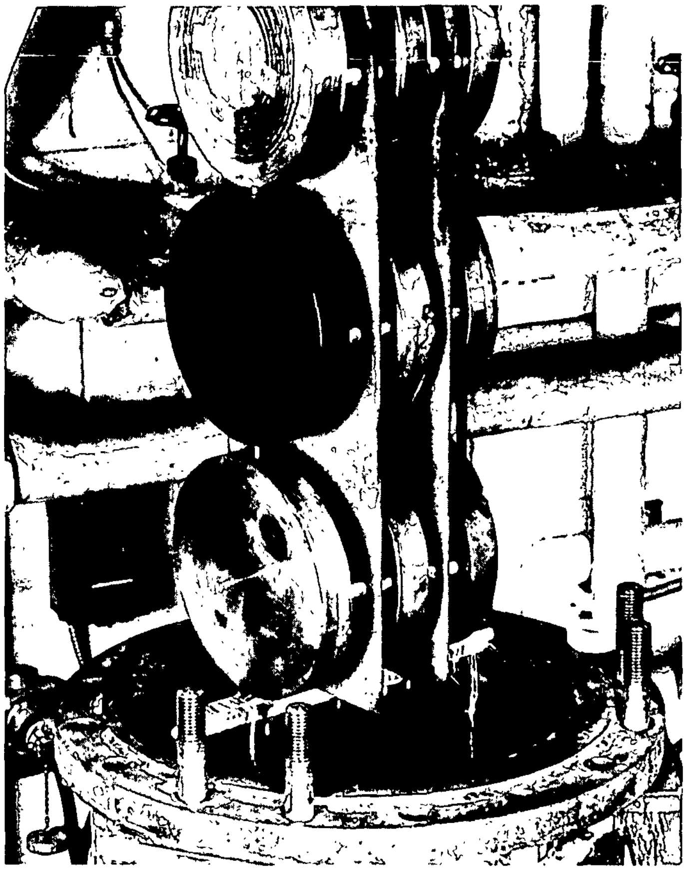


Figure 25: Photograph of the CPTW specimens being removed from the pilot plant autoclave for examination.

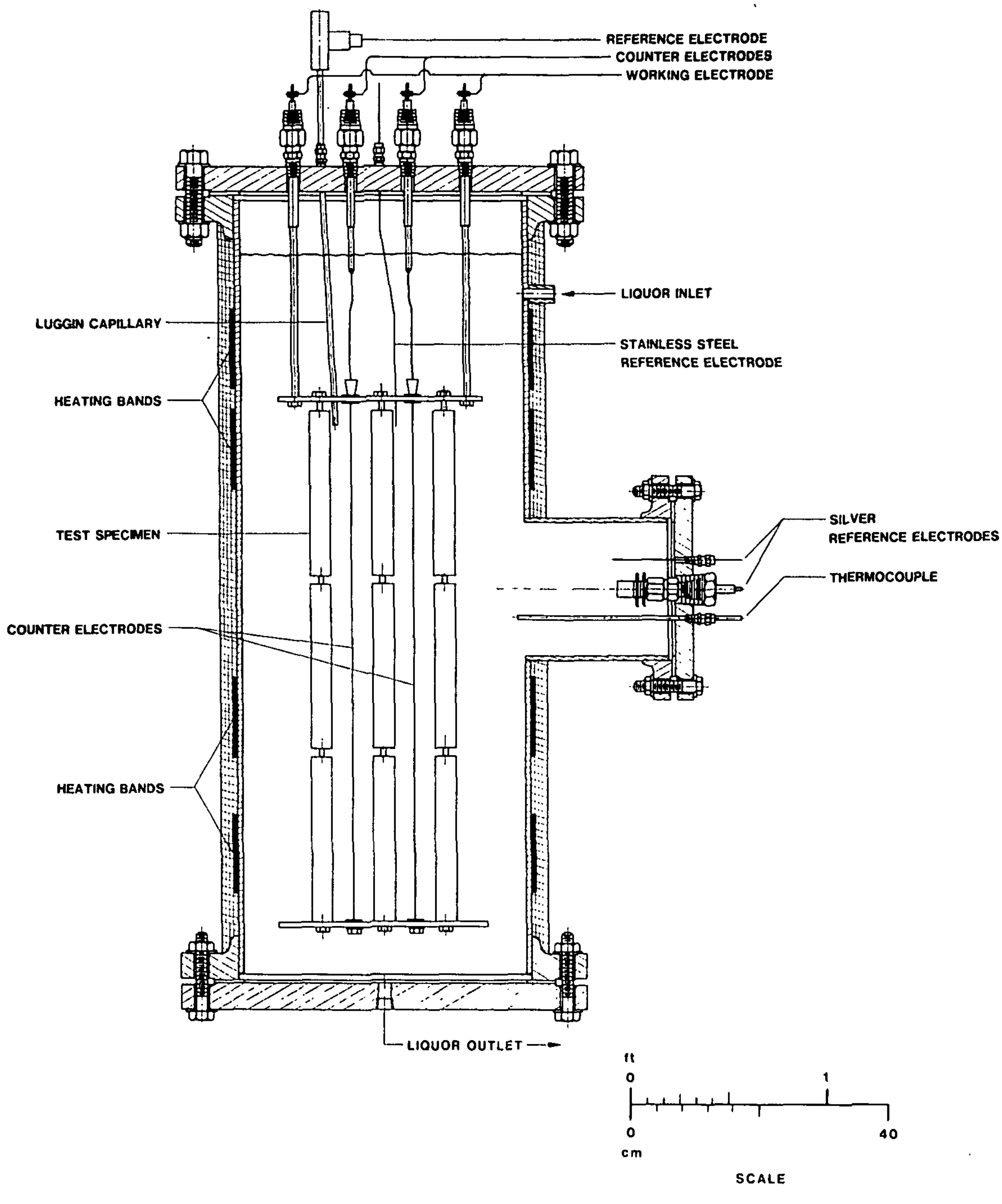


Figure 24: Schematic showing the arrangement of the specimens and counter-electrodes inside the 200L pilot plant autoclave. From left to right, starting with the top row, the specimens were: Inconel 82 overlay, temper-bead weld, 309 SS overlay, worst-case weld, E6010 capping pass, sealed thermal spray, stress relieved, shotpeened, control.

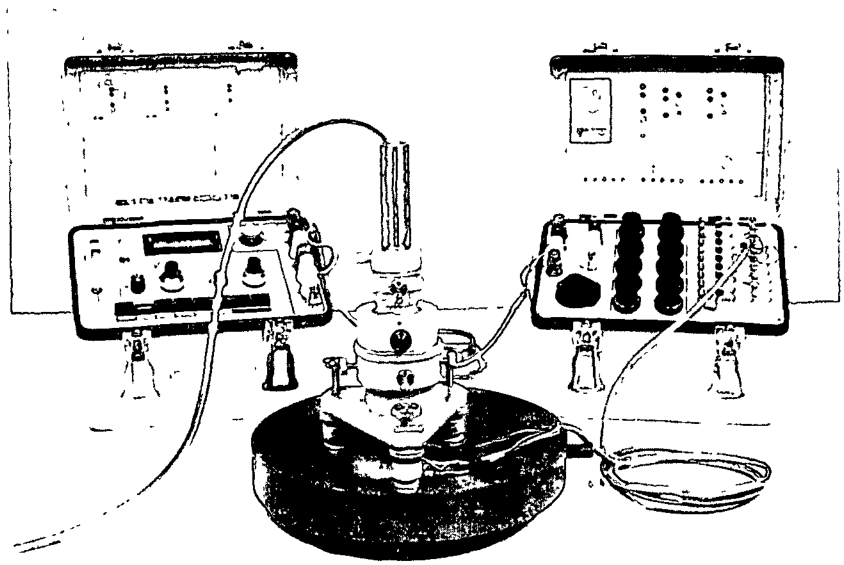


Figure 23: Photograph showing the residual stress measurement equipment. The drill and alignment jig are mounted on a CPTW specimen in the foreground. The bridge and balance units are in the background.

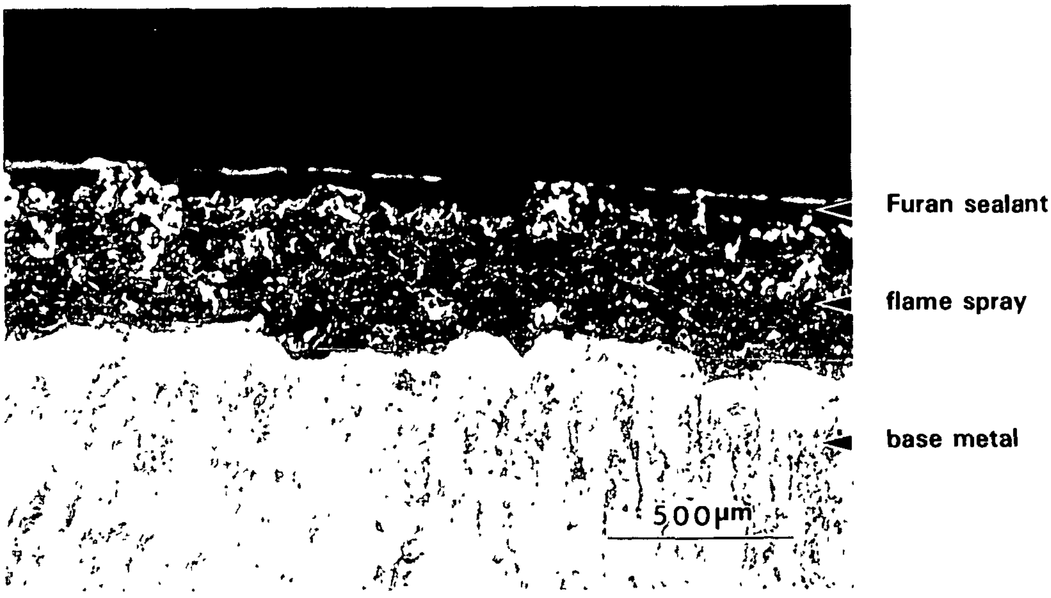


Figure 21: Micrograph of a polished and etched section cut from the thermal spray specimen showing the porosity of the flame spray coating. The sealant is the dark layer at the top of the photograph and the flame spray coating is visible in the centre of the photograph.

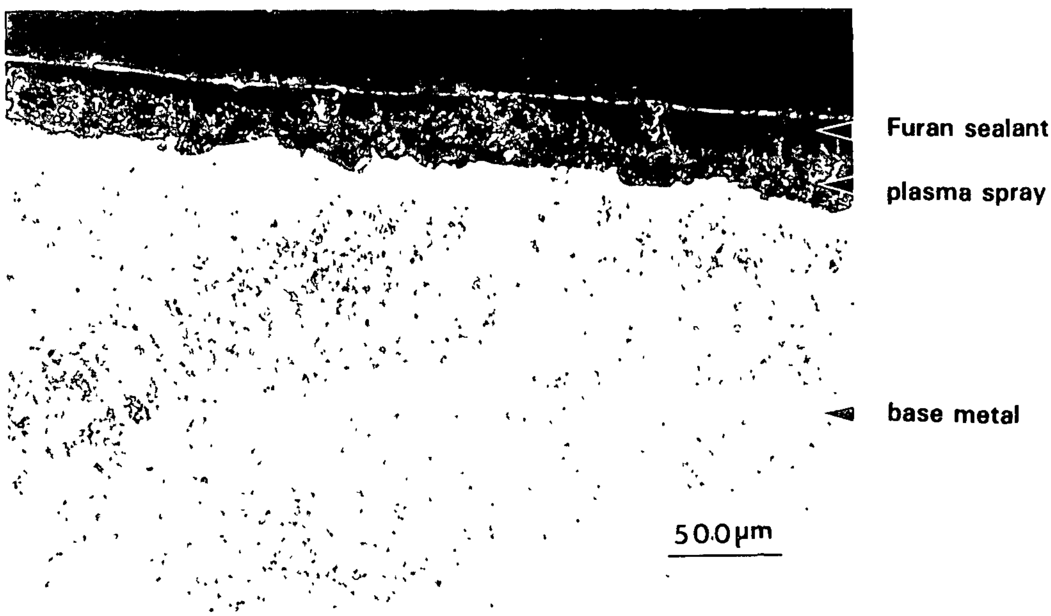


Figure 22: Micrograph of a polished and etched section cut from the plasma sprayed side of the specimen. The sealant is near the top of the photograph, while the plasma layer is in the centre of the photograph. Note that the plasma spray coating is thinner and less porous than the flame spray coating.

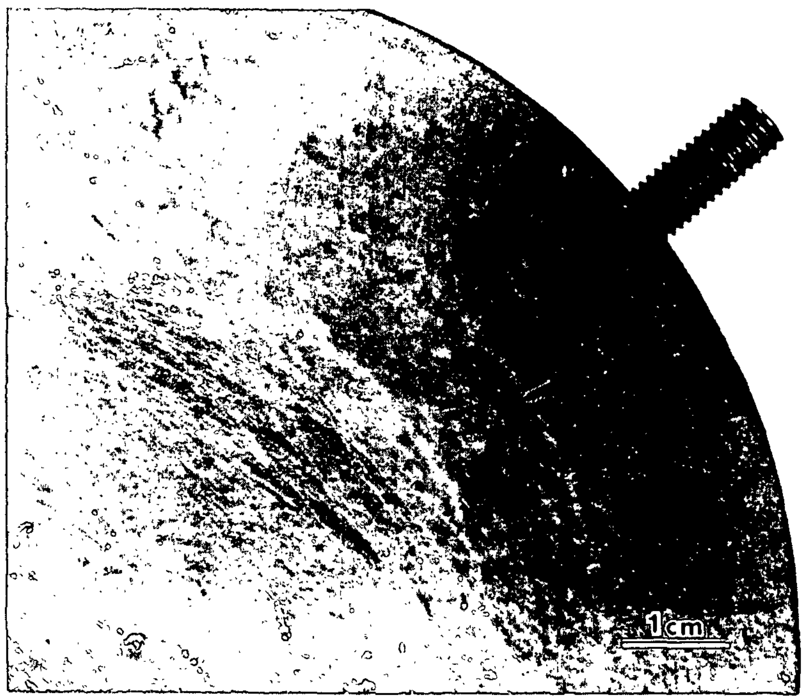


Figure 20: Photograph of the specimen protected from cracking by sealed thermal spray coatings. The Furan sealant is still in fairly good condition after 150 days testing at 115 C.

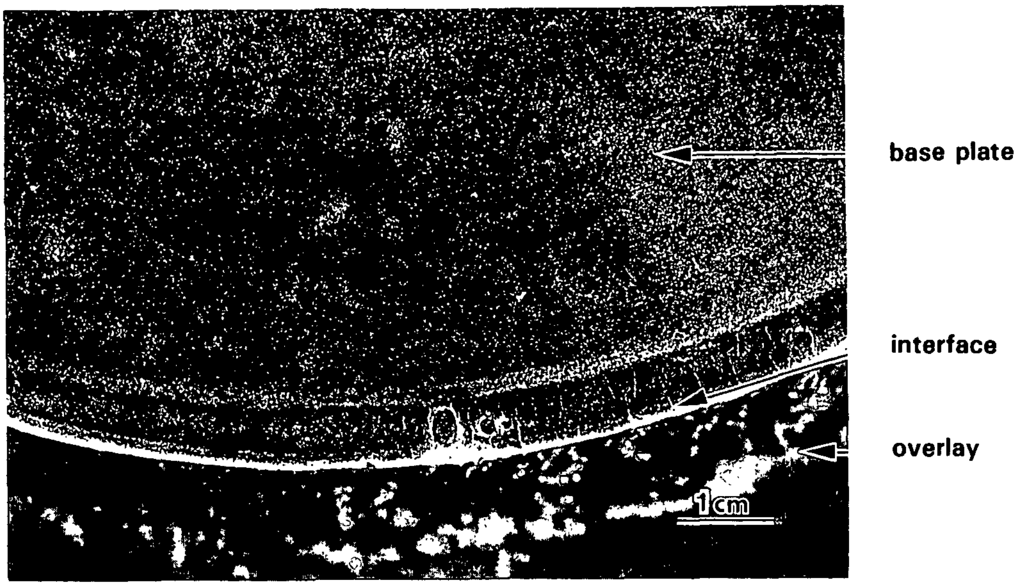


Figure 19: WFMP inspection showing short transverse cracks found at the inside circumference overlay/base metal interface of the 309 SS overlay specimen. The bright line around the circumference of the weld overlay is due to excess fluorescent material, and in this case does not indicate the presence of a crack.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	-125 (-18)	-155 (-23)	
B	-55 (-8)	-150 (-22)	AFTER EXPOSURE
C	170 (25)	30 (4)	
D	250 (36)	220 (32)	
E	-85 (-12)	-95 (-14)	
F	-210 (-30)	-285 (-41)	

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	40 (6)	-140 (-20)	
B	250 (36)	235 (34)	

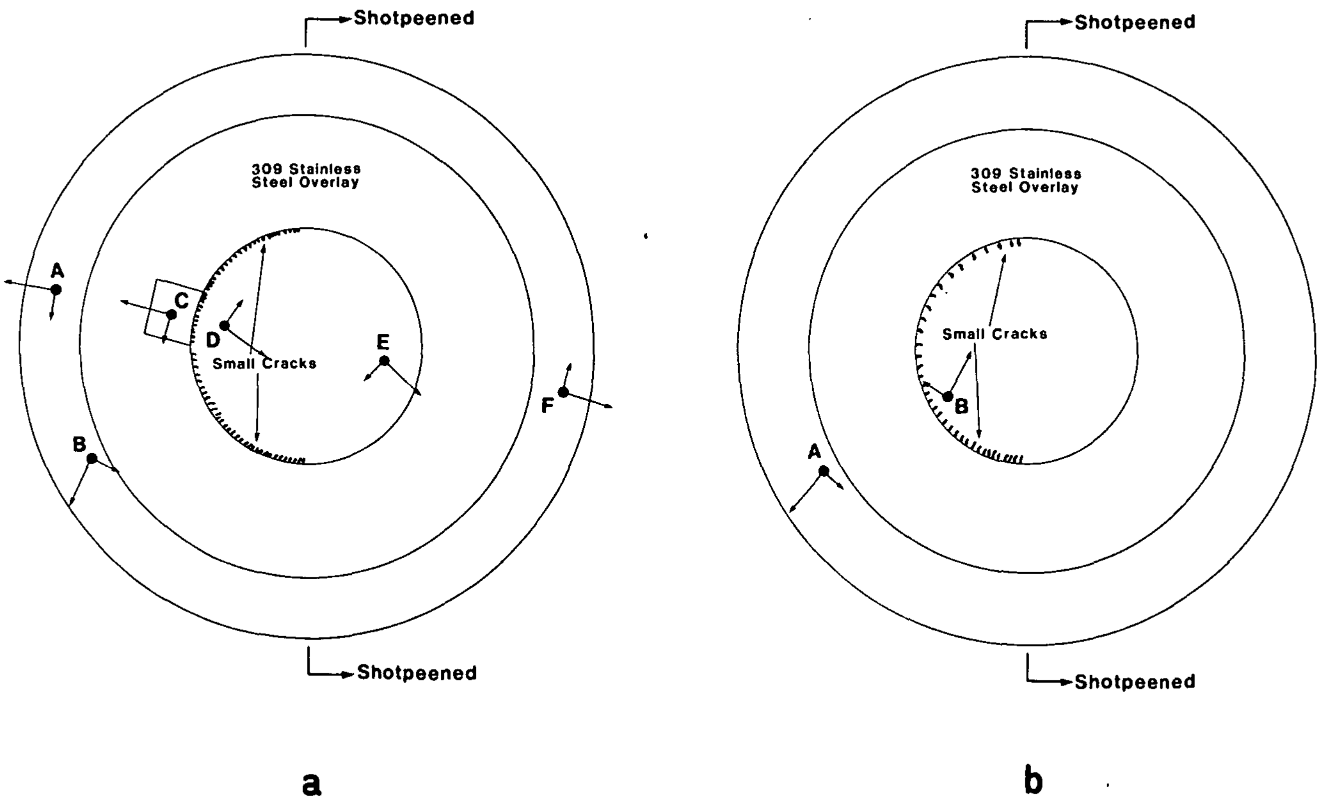
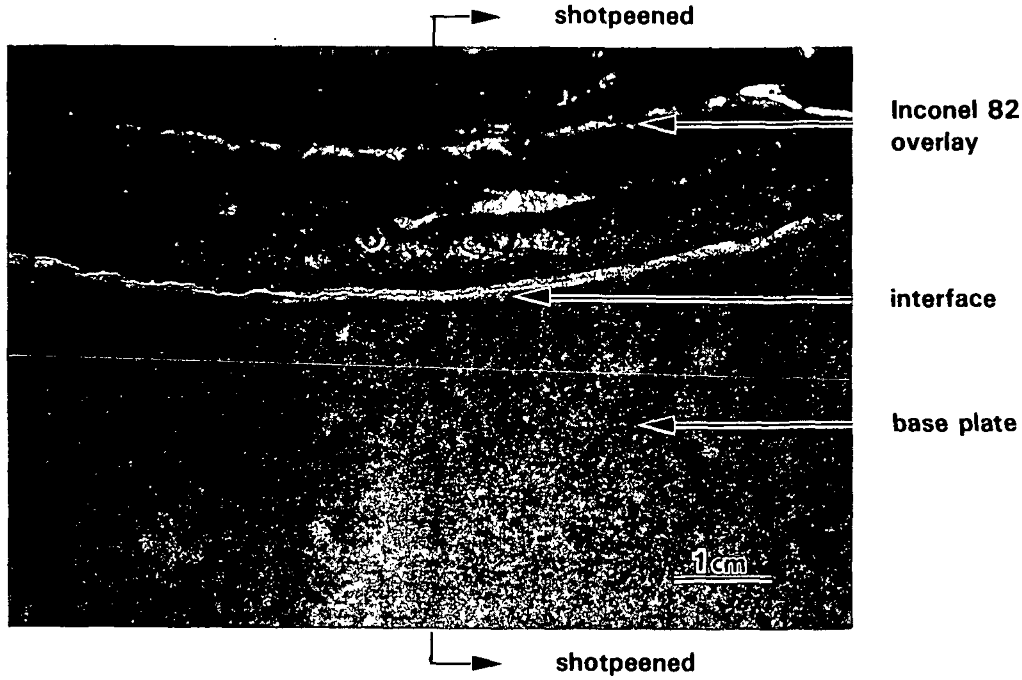


Figure 18: A map of cracks found on the 309 SS weld overlay specimen. Small transverse cracks were found on base plate immediately adjacent to the inside circumference the weld overlay on both faces. No cracking occurred on the shotpeened section of the specimen. Dots indicate points where residual stresses were measured. Long arrows indicate the orientation of the maximum measured stress; short arrows indicate the orientation of the minimum measured stress at each point. a) first face welded. b) second face welded.



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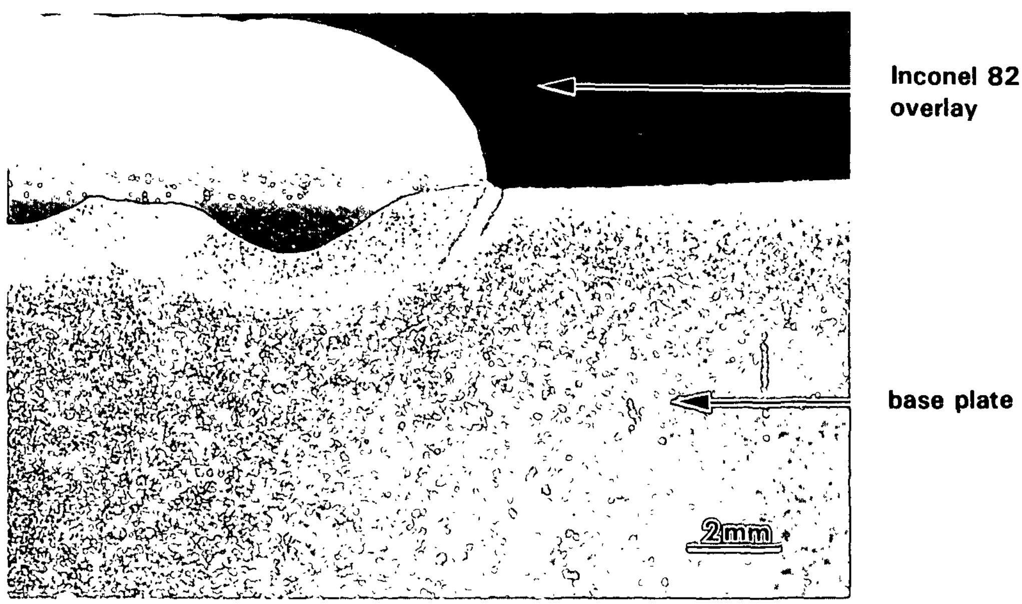
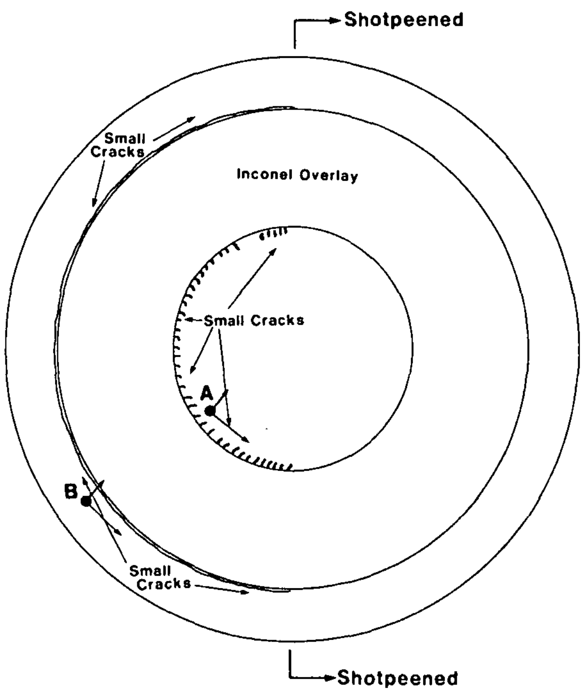


b

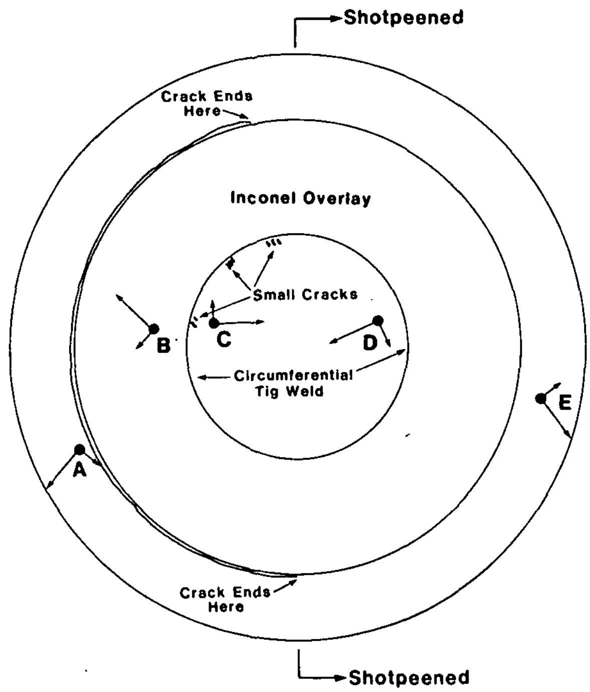
Figure 1.7: Deep circumferential cracking was found at the outer interface between the Inconel 82 weld overlay and the underlying carbon steel plate. a) WFMP inspection of an area at the outer interface of the Inconel 82 weld overlay (top of photo) and the underlying base plate (bottom of photo). b) Cross-section from the outer overlay/base metal interface showing the depth of crack penetration into the base metal.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	240 (34)	220 (32)	AFTER EXPOSURE
B	20 (3)	-210 (31)	AFTER EXPOSURE



a

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	210 (30)	-110 (-16)	
B	100 (15)	50 (7)	
C	130 (19)	90 (13)	
D	-100 (-15)	-150 (-21)	
E	-50 (-7)	-130 (-19)	



b

Figure 16: A map of cracks found on the Inconel 82 overlay specimen. Note that no cracking occurred on the shotpeened section of the specimen. Dots indicate points where residual stresses were measured. Long arrows indicate the orientation of the maximum measured stress; short arrows indicate the orientation of the minimum measured stress at each point. a) first face welded. b) second face welded.

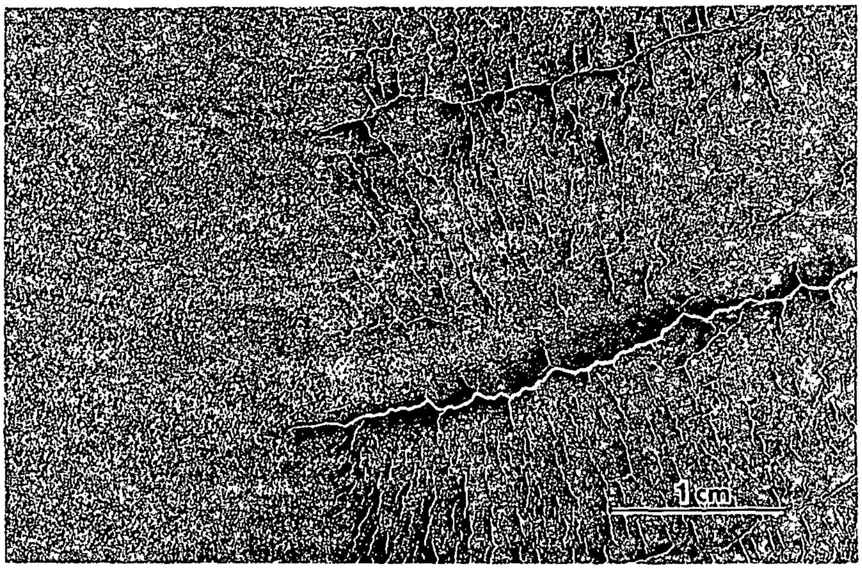


Figure 15: WFMP inspection of an area of the shotpeened specimen. The shotpeened section can be clearly seen on the left of the photograph. One circumferential crack has penetrated partially into the shotpeened area.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	650 (94)	140 (20)	
B	430 (62)	155 (22)	
C	-70 (-10)	-290 (-42)	

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	390 (57)	235 (34)	AFTER EXPOSURE

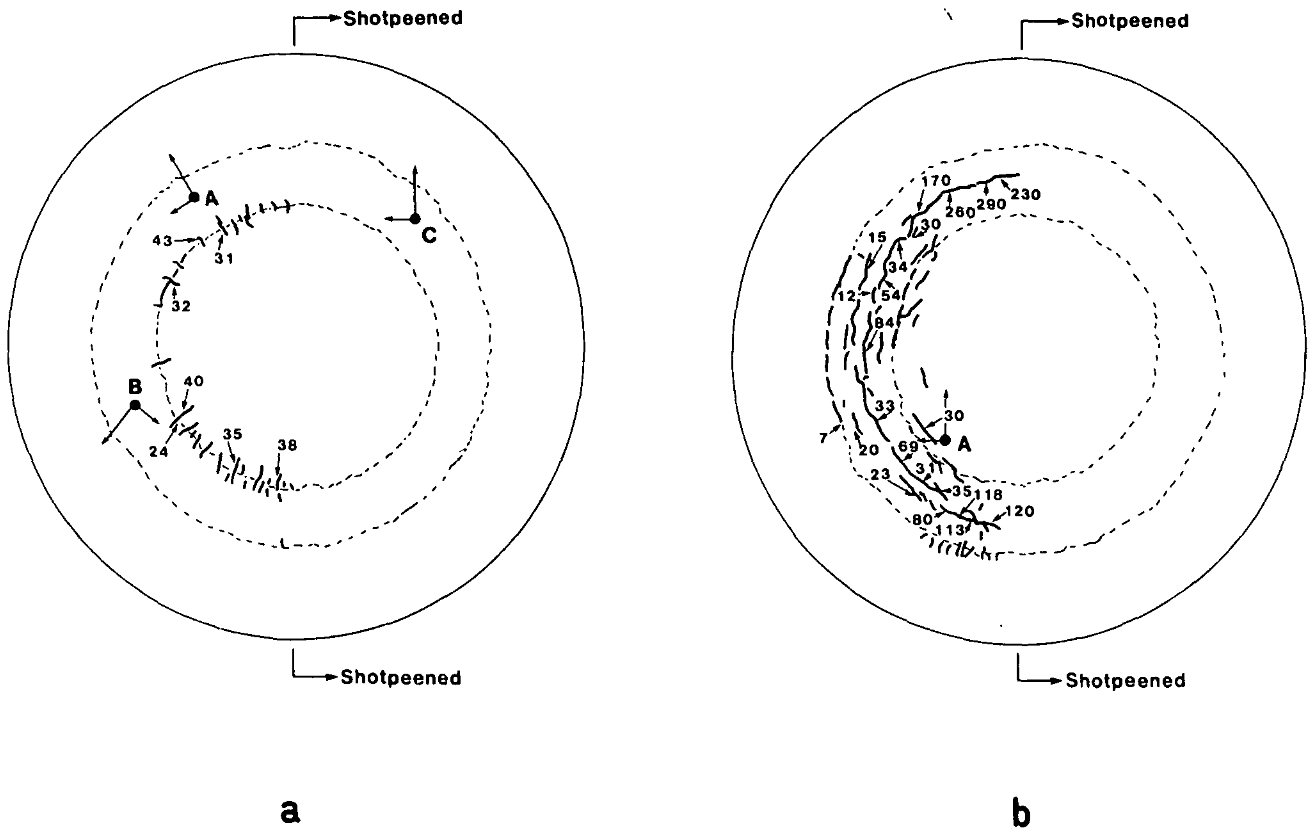


Figure 14: A map of cracks found on the shotpeened specimen. Crack propagation occurred only on the non-peened sections of the specimen. Crack depths were measured with the TSI crack depth indicator and are given in thousands of an inch. Dots indicate points where residual stresses were measured. Long arrows indicate the orientation of the maximum measured stress; short arrows indicate the orientation of the minimum measured stress at each point. a) first face welded. b) second face welded.

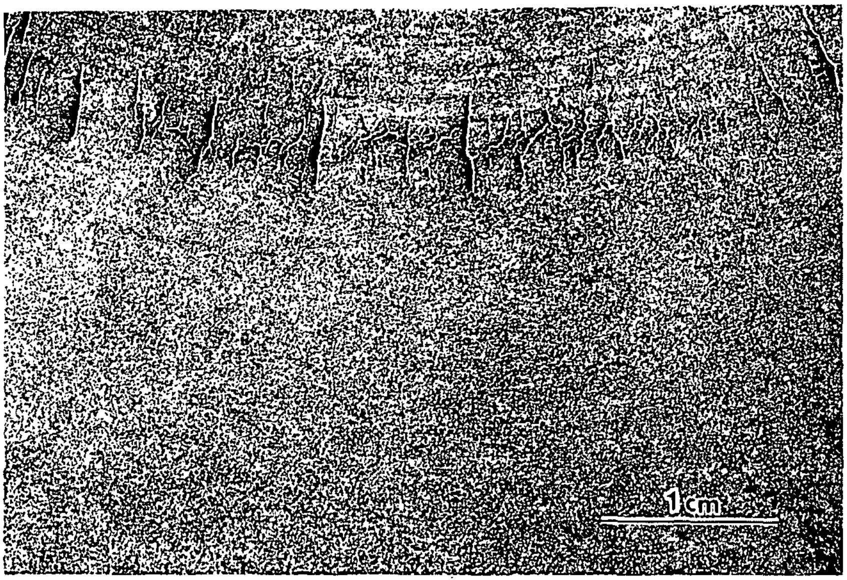
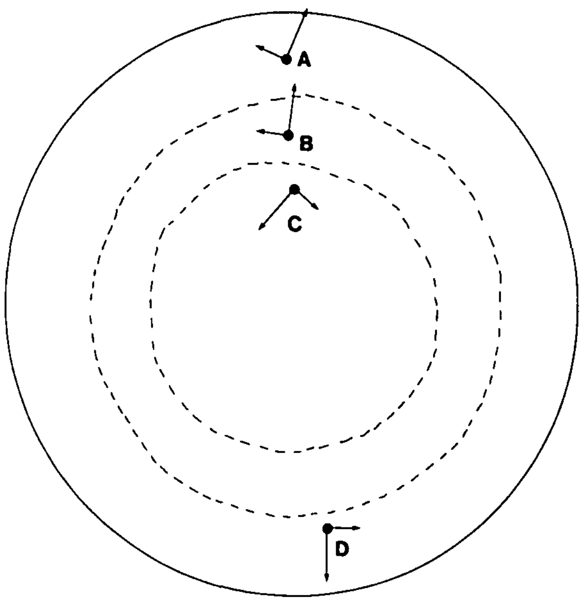


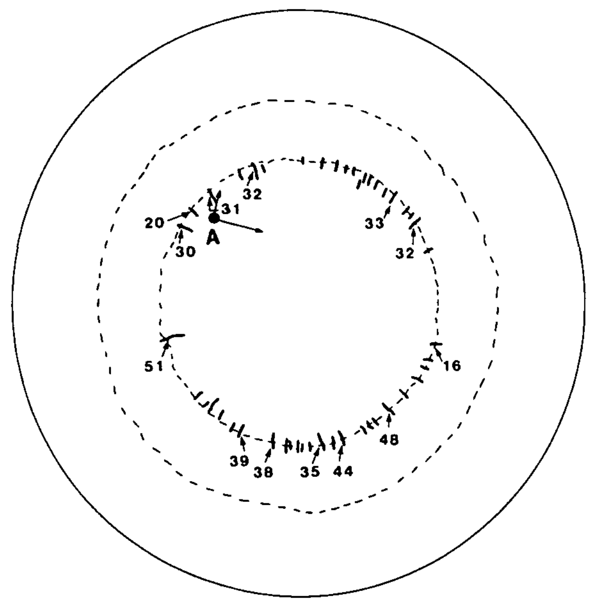
Figure 13: WFMP inspection of an area of the temper-bead welded specimen. Cracking occurred only in a single pass along the inside circumference of the weld.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	-40 (6)	-85 (-12)	
B	200 (29)	25 (4)	
C	115 (17)	85 (12)	
D	50 (7)	-5 (-1)	



a

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	230 (33)	170 (25)	AFTER EXPOSURE



b

Figure 12: A map of cracks found on the temper-bead welded specimen. Crack depths were measured with the TSI crack depth indicator and are given in thousands of an inch. Dots indicate points where residual stresses were measured. Long arrows indicate the orientation of the maximum measured stress; short arrows indicate the orientation of the minimum measured stress at each point. a) first face welded. b) second face welded.

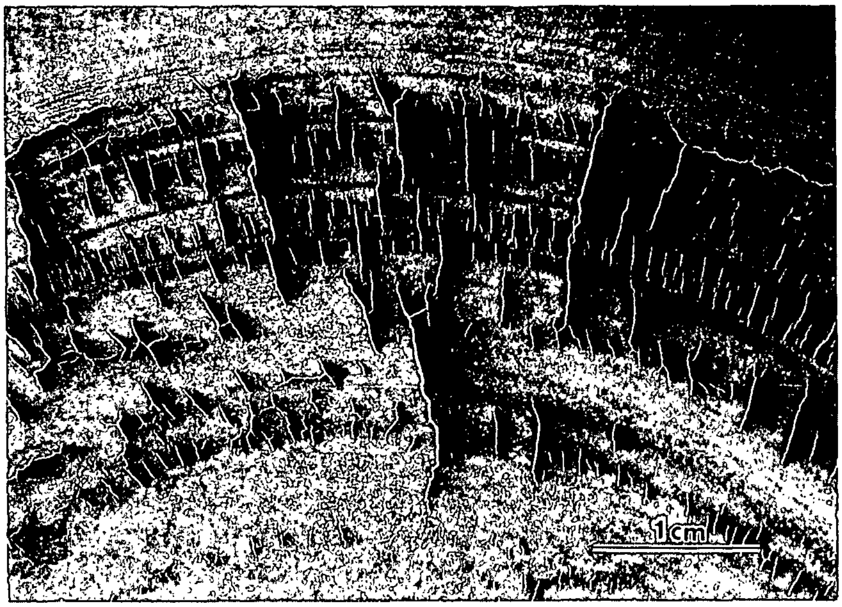
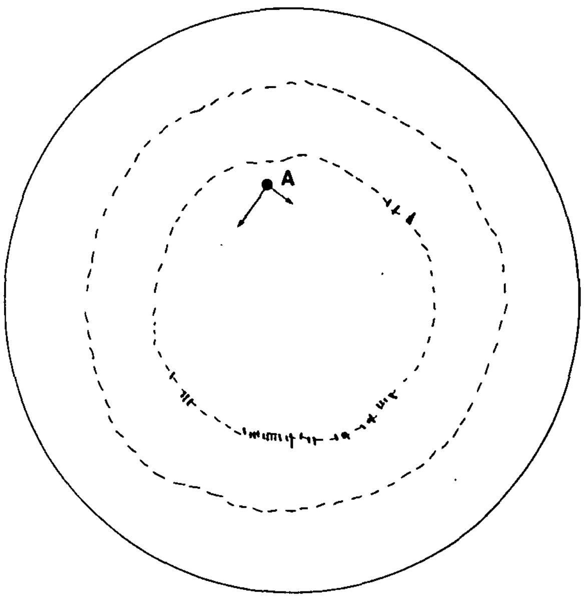


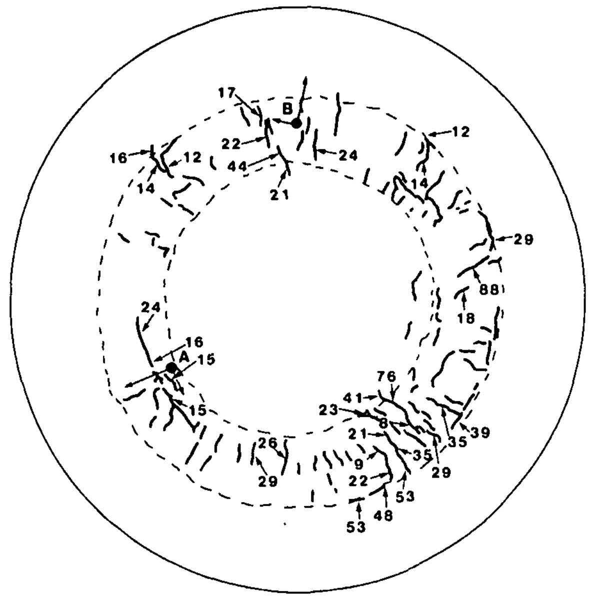
Figure 11: WFMP inspection of an area of the specimen welded with a 6010 capping pass. Note that the cracks are predominantly transverse to the weld.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	245 (36)	170 (25)	AFTER EXPOSURE



a

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	325 (47)	310 (45)	
B	575 (83)	180 (26)	



b

Figure 10: A map of cracks found on the specimen made with a 6010 capping pass. Crack orientation is primarily transverse to the weld. Crack depths were measured with the TSI crack depth indicator and are given in thousands of an inch. Dots indicate points where residual stresses were measured. Long arrows indicate the orientation of the maximum measured stress; short arrows indicate the orientation of the minimum measured stress at each point. a) first face welded. b) second face welded.

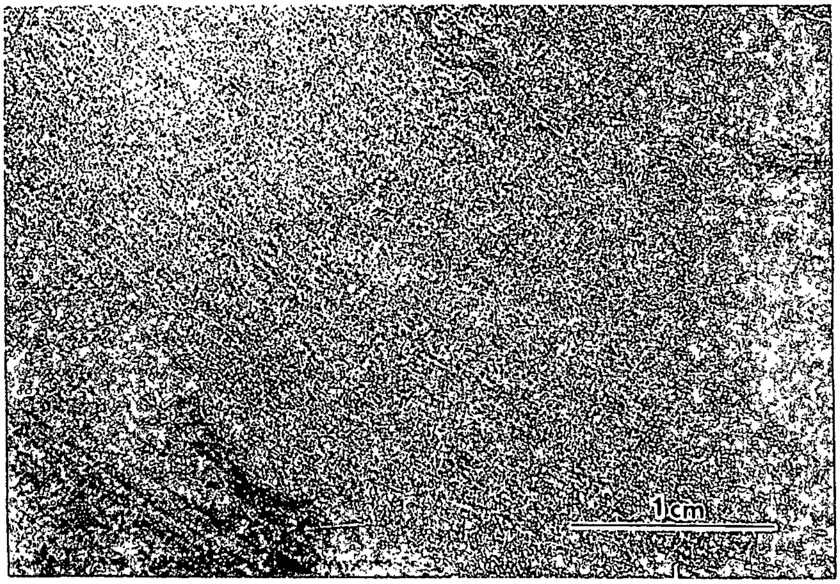
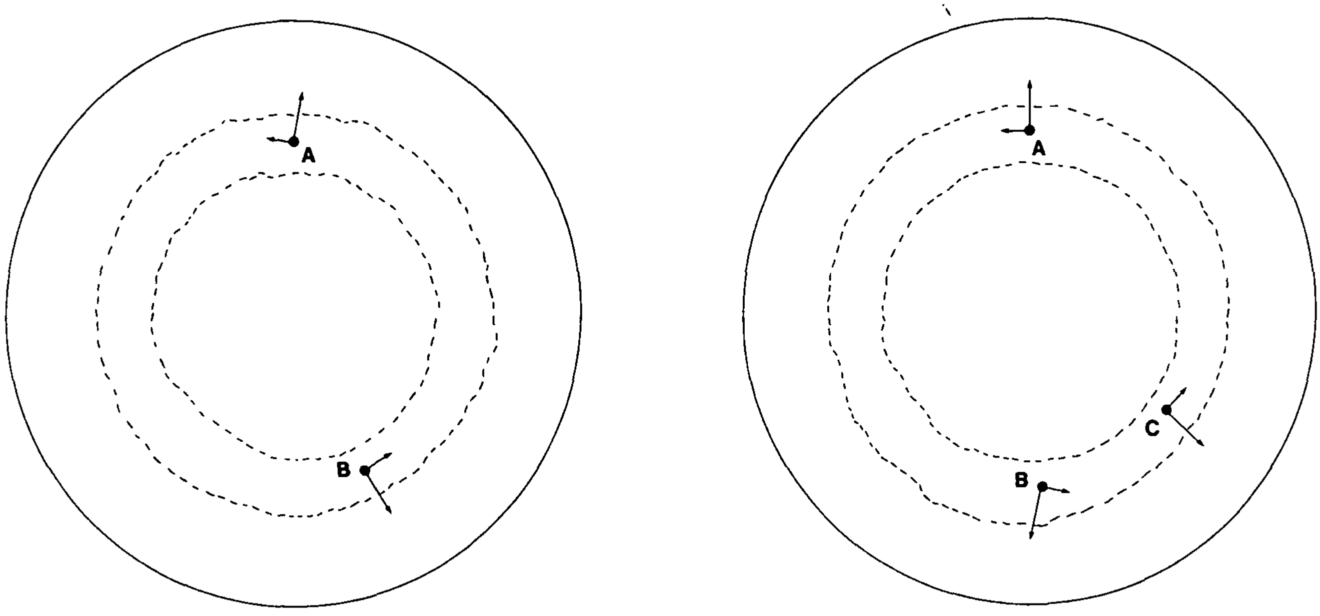


Figure 9: No cracks were revealed by WFMP inspection of the stress relieved specimen.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	380 (55)	360 (52)	BEFORE STRESS RELIEF
B	20 (3)	-20 (-3)	AFTER STRESS RELIEF

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	230 (33)	-5 (-1)	BEFORE STRESS RELIEF
B	-10 (-1)	-30 (-4)	AFTER STRESS RELIEF
C	1 (0.1)	-30 (-4)	AFTER STRESS RELIEF



a b

Figure 8: No cracks were found on the stress relieved specimen. Dots indicate points where residual stresses were measured. Long arrows show the direction of the maximum stress measured; short arrows the direction of the minimum stress measured at each point. a) first face welded. b) second face welded.

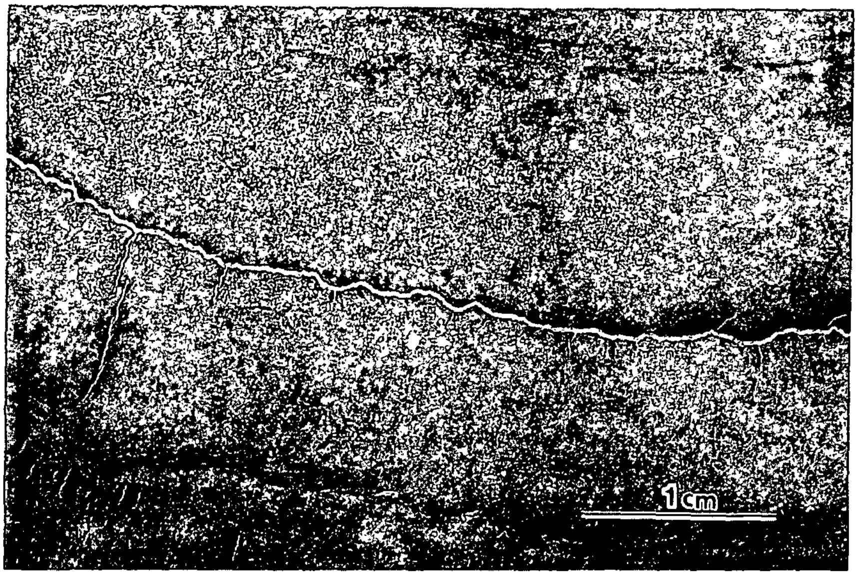
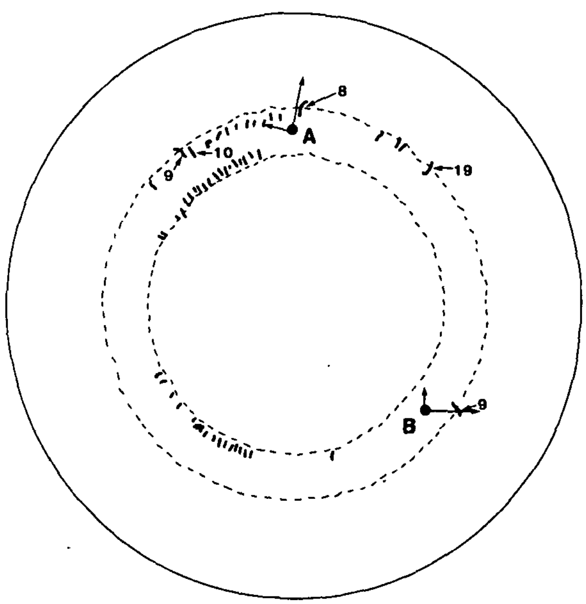


Figure 7: WFMP inspection of an area of the specimen welded with the worst-case welding procedure. The single circumferential crack penetrated as much as 12 mm (0.5 in) into the specimen and was easily visible with the naked eye.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	485 (70)	345 (50)	
B	300 (43)	50 (7)	AFTER EXPOSURE

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	425 (62)	155 (23)	AFTER EXPOSURE
B	515 (75)	365 (53)	BEFORE EXPOSURE
C	145 (21)	-20 (-3)	AFTER EXPOSURE



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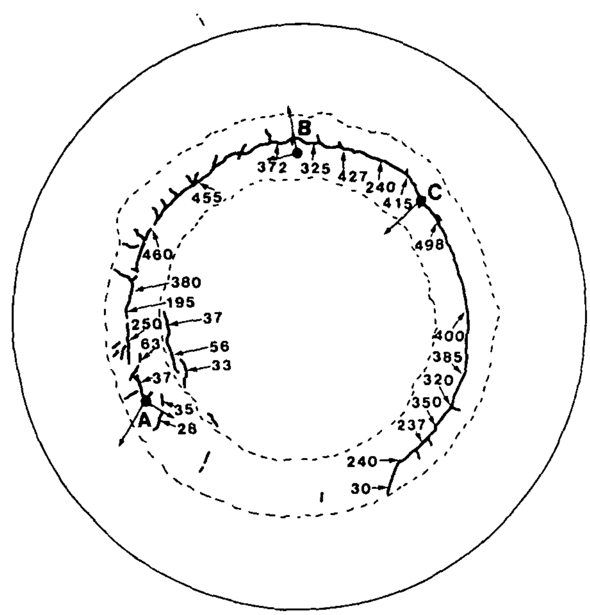

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Figure 6: A map of cracks found on the specimen welded with the worst-case welding procedure. Crack depths were measured with the TSI crack depth indicator and are given in thousands of an inch. Dots indicate points where residual stresses were measured. Long arrows indicate the orientation of the maximum measured stress; short arrows indicate the orientation of the minimum measured stress at each point. a) first face welded. b) second face welded.

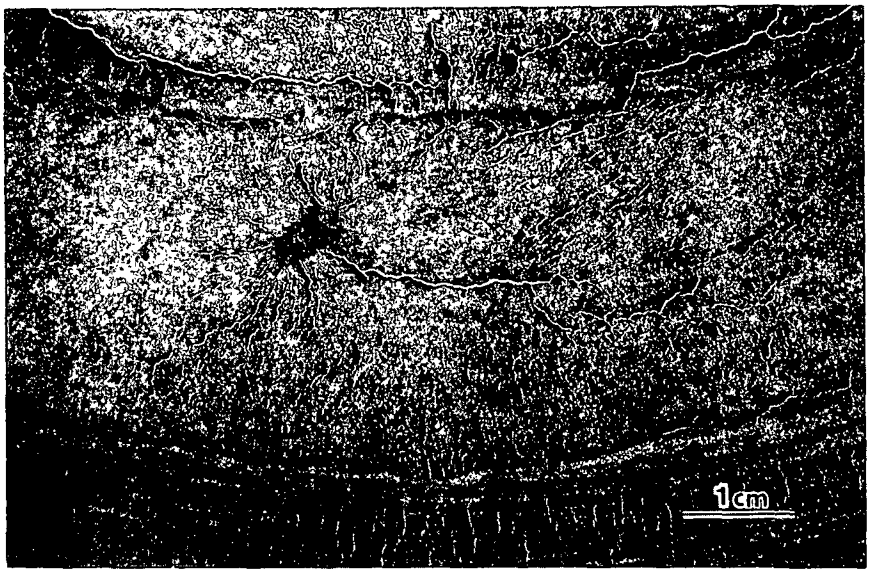
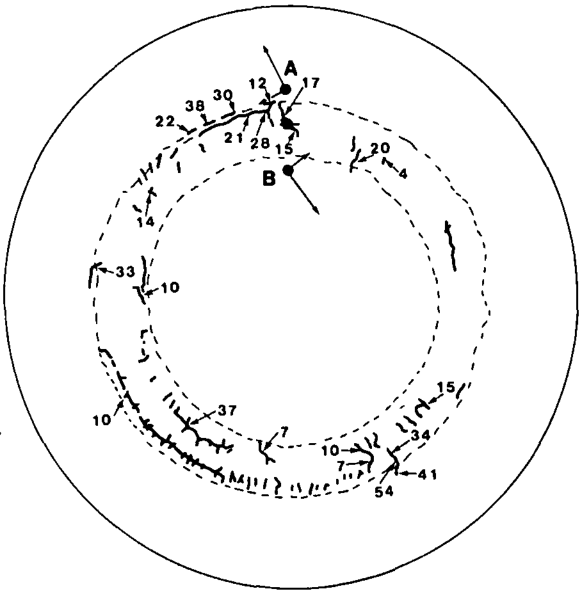


Figure 5: WFMP inspection of an area of the standard control specimen. SCC occurred in both the weld metal and in the weld HAZ.

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	360 (52)	135 (20)	
B	335 (49)	145 (21)	

	MAX STRESS MPa (ksi)	MIN STRESS MPa (ksi)	COMMENTS
A	102 (15)	-235 (-34)	
B	299 (43)	-7 (-1)	



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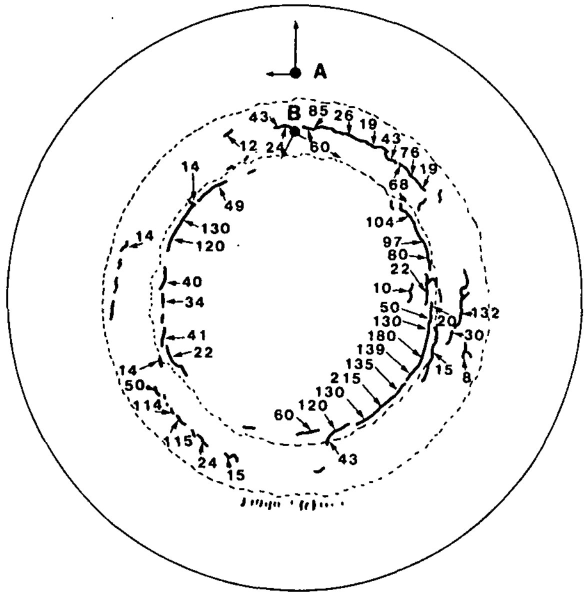


b

Figure 4: A map of cracks found on the control specimen. Crack depths were measured with the TSI crack depth indicator and are given in thousands of an inch. Dots indicate points where residual stresses were measured. Long arrows indicate the orientation of the maximum measured stress; short arrows indicate the orientation of the minimum measured stress at each point. a) first face welded. b) second face welded.

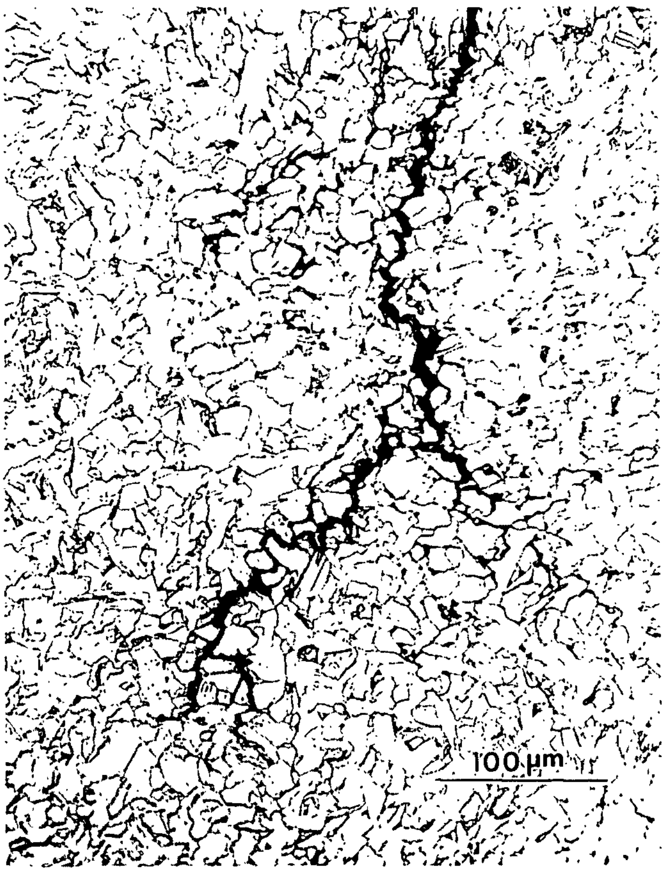
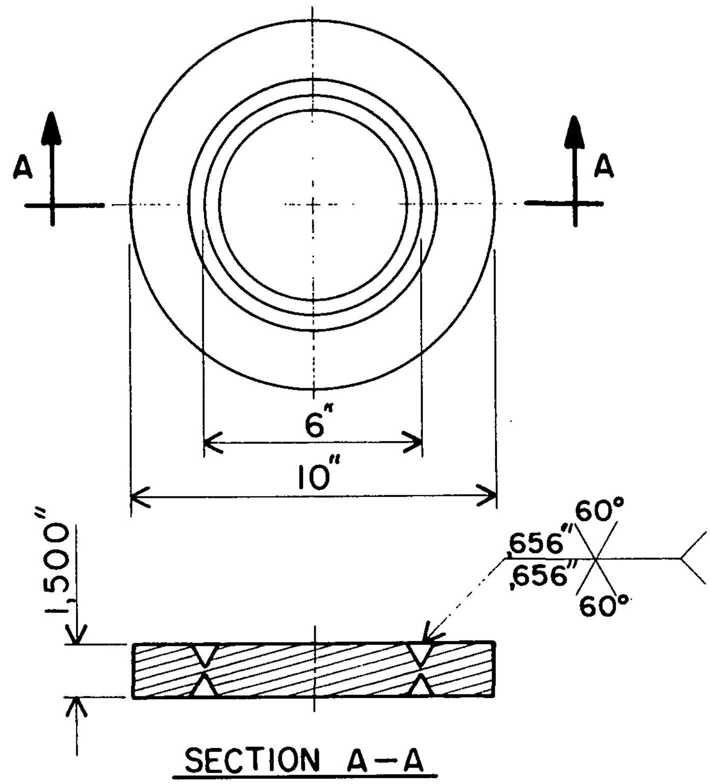
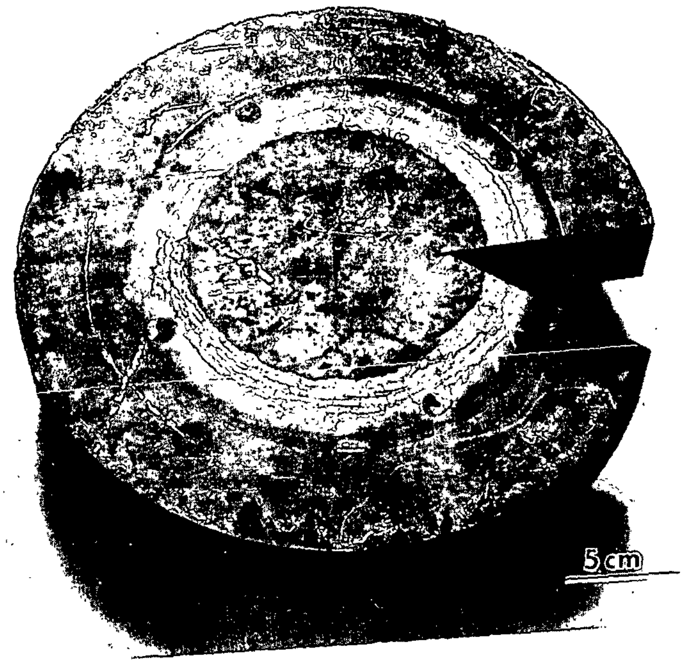


Figure 3: Micrograph showing the typical intergranular crack path of caustic stress corrosion cracking. This cross-section, showing recrystallized weld metal, was cut from the control specimen. Crack propagation was from the top to the bottom of the photograph. The appearance of cracks in the other specimens was similar.

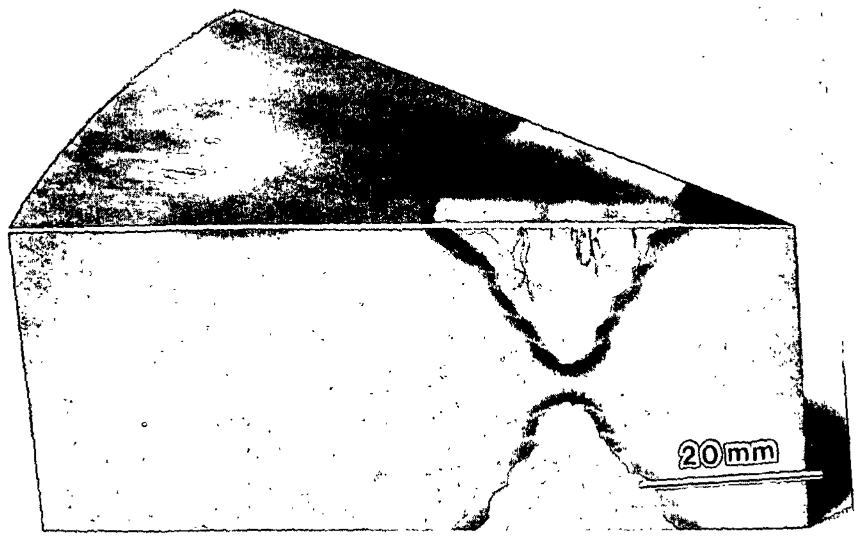


14		CIRCULAR PATCH WELD SPECIMEN		
ITEM	QTY	DESCRIPTION		MATERIAL OR PART NO.
26·02·86	P. KNEE			MAT'L:
DATE	DRAWN	APP'D	REV.	CORROSION SPECIMENS
PULP & PAPER RESEARCH INSTITUTE OF CANADA			SCALE 1–4	DWG. NO: A-8301-A-11

Figure 2: Schematic showing the design of the CPTW specimens.



a



b

Figure 1: Preliminary testing showed that CPTW specimens would crack readily in 33% NaOH at a worst-case cracking potential. a) CPTW specimen after testing, with segment cut-out for inspection. b) Segment removed from CPTW specimen shown in Figure 1a. The welds on the cross-section of this segment have been revealed by etching. Stress corrosion cracks can be seen penetrating deeply into the top weld. The experiment duration was 7 days.

TABLE II: Summary of Visual Inspections and Apparent Crack Depths(1) in CPTW Specimens.

specimen	Visual inspection results	avg. crack depth mm (in)	max. crack depth mm (in)
control	numerous circumferential cracks in weld and HAZ on both sides.	A: 0.5 [illegible] B: 1.[illegible]	[illegible] 3.4 [illegible]
worst-case weld	minor cracking on "A" side. severe circumferential weld crack on "B" side.	A: 0.3 [illegible] B: 6.4 [illegible]	[illegible].5 [illegible] [illegible]
stress relieved	no cracking.	0.0	0.0
6010 capping pass	minor cracking on "A" side. many transverse cracks on "B" side.	A: ---[illegible] B: 0.7 (0.03)	---[illegible] 2.2 (0.[illegible]
temper-bead welded	no cracking on "A" side. minor transverse cracks on inside weld diameter on "B" side.	A: 0.0 B: 0.8 (0.03)	1.3 (0.[illegible]
shotpeened	no cracks in shotpeened portions of specimen.	0.0	0.0
Inconel 82(2)	circumferential cracks on both sides at outer base plate/overlay interface.	A: --- B: ---	0.5 (0.[illegible] 6.0 (0.[illegible]
309 SS	minor transverse cracks at inner base plate/overlay interface only.	---(3)	---(3)
sealed thermal sprays	no cracking. minor degradation of sealant.	0.0	0.0

NOTES: 1) as measured by TSI Model CC 800B crack depth indicator.

2) depth of outer circumferential crack in Inconel weld overlay specimen could not be measured by the crack depth indicator. Crack depth is given for a single measurement at a specimen cross-section.

3) small, superficial cracks which could not be measured by the crack depth gauge.

TABLE I: Comparison of Actual Crack Depth Measurements[1] versus TSI Model CC 800B Crack Depth Indicator.

specimen	Model CC 800B mm (in)	Actual Crack Depth mm (in)
control	0.9 (0.04) 3.4 (0.13)	1.8 (0.07) 3.0 (0.12)
worst-case weld	11.7 (0.46) 11.6 (0.46)	10.0 (0.39) 13.0 (0.51)
stress relieved	0.0	0.0
6010 capping pass	1.3 (0.05) 1.9 (0.08)	1.8 (0.07) 3.2 (0.13)
temper-bead weld	0.9 (0.04) 1.2 (0.04)	2.4 (0.09) 2.4 (0.09)
shotpeened[2]	1.0 (0.04) 6.6 (0.26)	0.9 (0.04) 4.1 (0.16)
Inconel 82	---[3] ---[3]	0.6 (0.02)[4] 6.0 (0.25)[4]
309 SS	---[3] ---[3]	---[5] ---[5]
sealed thermal spray	0.0	0.0

NOTES: (1) for randomly chosen locations where specimens were sectioned. measurements are compared to closest TSI Model CC 800 B measurement made in vicinity of cut.
(2) for non-shotpeened half only.
(3) No measurements made with TSI Model CC 800 B.
(4) crack depths reported for circumferential cracks only.
(5) shallow transverse cracks, depth not measured.

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16) "Boiler and Pressure Vessel Code", Section VIII, Division 1, UW-40 and UCS-56, ASME, New York, NY., (1968).

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balanced Luggin capillary was inserted into the digester to measure the electrochemical potential of the specimens. The nominal liquor concentration used was 40 g/L NaOH, 20 g/L Na_2S and 20 g/L Na_2CO_3 and the liquor was regularly sampled and replaced as necessary. The liquor was constantly circulated through the vessel. Specimen exposure was for a total of 150 days at a temperature of 115 C. The specimens were removed from the digester for a brief inspection after approximately 100 days, and then returned for the remainder of the test period.

Inspection: All specimens were inspected for cracking by WFMP prior to exposure to the liquor. Following the test period, the specimens were cleaned with an inhibited HCl solution at room temperature to remove tightly adherent passive films on the surface. The specimens were then scrubbed with soap and water and dried. WFMP inspection was used to determine the extent of cracking on the surfaces of the specimens. Crack depths were measured with a Test Systems International model CC 800B crack depth indicator, using the model MP4-B four pin resistance probe. The accuracy of the crack depth measurements were checked by cutting wedges from each specimen and comparing actual crack depths against previously measured values (Table I). The integrity of the sealed thermal spray coatings was determined by visual examination of the specimen and subsequent examination of a cross-section under a light microscope.

IV. ACKNOWLEDGEMENTS

The help of Gary Zellmer (Metco Inc.) and Ernie Edgerly and his welding crew (Kamyr Installations) and the generous assistance of their respective companies with specimen preparation is gratefully acknowledged. Thanks also to MacMillan Bloedel Research for the loan of their crack depth detector. This work could not have been completed without the dedicated assistance of Neville Stead, Andre Menard, Stanley Rychel and Fred Herbert.

We would also like to acknowledge the support and encouragement of members of the Digester Cracking Research Committee during this work and to thank Chicago, Bridge and Iron Company, Houston (and R. Fuchs in particular) for supplying the steel used in these experiments.

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was plasma sprayed with Metco 465 powder, while the other face was flame sprayed with a Metco 444 coating. The edge of the specimen was also flame sprayed to provide a good, adherent surface for the organic sealant. A modified Furan sealant (Metco # CE 2236) was then thickly brushed by hand onto all surfaces and allowed to dry.

A standard test specimen and the weld overlay specimens were commercially shotpeened to evaluate the influence of shotpeening on SCC. One half of each face of the specimens was masked off, while the other half was peened. A nominal shot diameter of 0.230" was used on the exposed sections of the specimens, with an Almen shot intensity of 0.014/0.018 "A" and a coverage of 125%. The notch at the interface between the weld overlays and the carbon steel plate was shotpeened with 0.110" nominal size steel shot with an Almen intensity of 0.010/0.012 "A" and a 125% coverage.

Residual Stress Measurements: A blind hole drilling method was used to measure residual surface stresses in the test specimens [12]. A Measurements Group RS-200 milling guide and high speed (400,000 rpm) air turbine were employed, along with a Model P-350A strain indicator and SB-1 switch and balance unit (Figure 23). Measurements Group EA-06-062RE-120 or TEA-06-062RK-120 strain gage rosettes were used throughout the work and SS White FG dental carbide burrs were used to drill the holes.

Before residual stresses were measured, the surface at the desired location was prepared to a 600 grit finish. The smoothed area was then thoroughly cleaned and a strain gage rosette cemented onto the specimen. After connecting the rosette to the strain indicator through the switch and bridge unit, the milling guide was positioned over the rosette and a hole drilled through the centre of the rosette in 0.005" increments. After each increment, the drill was stopped and the relieved strains measured. In most cases, the majority of the residual stresses were relieved in the first three or four increments. The nominal hole diameter was 1.6 mm (0.062") and the maximum hole depth drilled was 1.3 mm (0.050"). Measured strain values were converted to residual stress by conventional analyses [12]. All residual stress values reported are the total stress relieved after drilling a hole depth of 1 mm (0.040").

Note that residual stress measurements of this type are generally considered to be accurate to within about ±10% [24,25]. As the residual tensile stresses approach the yield strength of the material (>70%), the accuracy of the measurement decreases due to plastic deformation around the drilled hole. The precision of the residual stress measurement technique was determined by measuring the residual stress present in a block of A516 Grade 70 steel from which residual stresses had been removed by sealing it in foil, heating it to 700 C (1300 F) for 24 hours, and then furnace cooling. Residual tensile stresses measured in the test block were less than 6 MPa (0.8 ksi).

Exposure to Liquor: A 200 L pilot plant digester, previously used for fracture mechanics stress corrosion testing in the TAPPI/IPC research program [3], was modified to accept nine of the CPTW specimens (Figures 24, 25). After all specimens had been prepared, and residual stress measurements completed, the specimens were placed in the digester. A stainless steel sheet counterelectrode was placed between each column of specimens, and the digester was also used as a counterelectrode to ensure even current density distribution over the specimens. A double junction Ag/AgCl reference electrode in a pressure-

[redacted]

were flame-cut from 38 mm (1.5") thick plates of the A516 Grade 70 steel. The blanks were then machined to a diameter of 254 mm (10") and opposing 152 mm (6") diameter grooves were cut into each face of the specimens (Figure 2).

Unless otherwise noted, each specimen was shielded metal arc (SMA) welded with 1/8" diameter E7018 weld electrodes using standard CBI welding procedures [3]. Specimens were preheated to 95 C (200 F) before welding and the maximum weld interpass temperature was restricted to 150 C (400 F). Air-blast cooling was employed to speed cooling between weld passes. Each groove was filled in with between 15 and 18 weld passes. During welding, the specimens were restrained to prevent distortion (and relief of residual stresses) by tack-welding them to the workbench. Each specimen was first welded completely on one side, then on the other and the faces of the specimens were subsequently skimmed on a lathe to a smooth, planar profile.

The worst-case weld specimen was SMA welded as described above, except that no preheat was used, and the specimen was cooled to ambient temperature after each pass. The specimen with an E6010 capping pass was welded according to the standard procedure, except that the final two layers of weld (approximately 8 passes) were made with 1/8" E6010 welding electrodes.

One of the welded specimens was stress relieved following guidelines for post-weld heat treatment given in the applicable portions of the ASME Boiler and Pressure Vessel Code [16]. The specimen was wrapped in stainless steel foil to prevent excessive oxidation, and heated to 590 C (1100 F) for one and a half hours. It was then left in the furnace and allowed to cool slowly back to room temperature.

The temper-bead specimen was welded with a temper-bead procedure developed by Ontario Hydro [19]. The sides of the grooves were first buttered from bottom to top with 3/32" E7018 electrodes, then the weld was completed in a conventional manner with 1/8" E7018 electrodes, except that the last pass was finished with the final weld bead in the centre of the weld. In this way, all underlying weld beads were grain-refined by a subsequent weld bead and a soft, tempered microstructure was left in the heat-affected zones of the weld. In addition, the unrefined weld metal of the last bead is left over a soft, refined microstructure, so that there is no preferred path for crack propagation through the weld.

Inconel 82 and 309 SS weld overlays were applied by Kamyr Installations, Glens Falls, New York to previously prepared CPTW specimens. All weld overlay was applied by a metal inert gas (MIG) welding process using standard Kamyr procedures. Inconel 82 wire was supplied by Huntington Alloys (heat #NX 99C8) and 309L SS wire was HISI Unibraze (heat #8E 16759). The specimens were mounted horizontally on a rotating pedestal and an automatic welding head was used to apply the overlay. Specimens were preheated to 95 C (200 F) and a maximum interpass weld temperature of 150 C (300 F) was maintained at all times. Approximately 8 passes were applied over the carbon steel weld on both faces of each specimen, starting on the outside diameter and working towards the centre of the specimen.

Flame and plasma spray coatings were applied by Metco. Inc., Westbury, New York. The test specimen was first prepared by blasting with Al_2O_3 fine grit and then finishing with G16 angular steel grit. One face of the specimen

SCC was found [17]. This test method therefore appears to provide a realistic assessment of cracking and crack prevention in digesters.

These tests graphically illustrate the role that weld quality can play in determining the severity of SCC. Adequate pre-heat and interpass weld temperatures are essential to prevent rapid cooling of the weld and the formation of hard, crack-susceptible microstructures. Although mixed success has been achieved in the field using welding techniques such as temper-bead welding and applying an E6010, or similar, weld capping pass, this work provides evidence that improved welding procedures can reduce the severity of SCC in digesters.

Of the techniques investigated, stress relief was demonstrated to be the most effective at preventing SCC, and is highly advisable for all new digesters. While local stress relief of repair welds has not always prevented further cracking, any reduction in residual tensile stresses would seem beneficial in reducing the severity of SCC. Sealed thermal spray coatings also prevented SCC, although the lifetime of the sealant in a digester could not be predicted from these tests. Since general corrosion occurs in most continuous digesters, shotpeening may not be a desireable preventative measure, even though no SCC occurred in shotpeened sections of specimens in these tests.

A major discovery of this work is that SCC is possible at the interface between carbon steel base plate and Inconel or 309L SS weld overlay. Whether or not similar cracking occurs in a digester will depend primarily upon the magnitude of the residual tensile stress in the shell at the edge of the overlay, and the electrochemical potential of the digester. Note that stresses as high as the yield strength have been measured at the interface between carbon steel and Inconel or stainless steel weld overlays laid onto large plates to simulate weld overlay bands in digesters [21]. Stresses of this magnitude were more than enough to cause SCC in these tests. While the distribution of residual stresses in the weld overlay specimens may not be the same as in a digester, initiation of SCC and limited crack propagation must at least be considered a possibility at the carbon steel/weld overlay interface in digesters which have weld overlay in place. The ultimate severity of cracking will depend on whether sufficient tensile stress can be sustained at the crack tip for continued propagation into the shell. This point may be important in the case of the weld overlays, since the region of high residual tensile stress would be expected to be quite shallow, compared to a digester shell weld.

Except for the specimen welded with the worst-case welding procedure, all of the CPTW specimens were prepared using the best possible practice of the respective crack prevention technique. It must be recognized that widely varying results can be obtained from the actual application of these techniques in a digester, and that their success or failure will depend on the quality of the application and the operating conditions of the particular digester.

III. EXPERIMENTAL

Specimen Preparation: Chemical composition and mechanical properties of the steel used in this work have been previously reported [3]. This steel is from the same heat as used by Paprican in previous research for the TAPPI/IPC digester cracking research program. Rough blanks for the CPTW specimens was also noticeably brittle after being exposed to the liquor. Some of the mechanical damage to the sealant may have occurred during handling of the specimen subsequent to exposure.

Micrographs of polished and etched sections cut from the specimen confirmed that the sealant had not separated from either surface over the duration of the test (Figures 21,22). They did show, however, that the Furan was extremely thin over high spots in the thermal spray coatings and, in a few places, did not cover the underlying material. The thickness of the Furan sealant was not measured prior to exposure, and therefore it could not be determined whether it had thinned during the exposure period. The micrographs also made it clear that the Metco 444 flame spray coating contained much more porosity than the Metco 465 plasma spray coating. Increased surface roughness (and porosity) would probably allow the sealant to bond better to the thermal spray coating. However, in the event that the sealant failed to completely cover the underlying thermal spray, greater porosity would also likely result in more rapid disbondment of the thermal spray coating.

Sealed thermal spray coatings have been extensively applied in only one continuous digester [22]. No cracking or disbondment has occurred in this digester since sealed thermal spray coatings were first applied in 1982 [23]. The results of the field trials, along with those reported in this paper, indicate that SCC of the digester welds can be prevented by the application of sealed thermal spray coatings. The mill experience has shown that penetration of liquor into an unsealed thermal spray coating will result in corrosion of the underlying carbon steel and subsequent rapid disbondment of the coating. For this reason, the organic sealant is the critical component of this crack prevention method.

The Furan sealant may deteriorate with exposure to kraft liquor at digester temperatures. However, after 150 days at 115 C, it still protected the underlying thermal spray coatings from disbondment and the carbon steel weld from SCC in the experiment reported here. The eventual service life of the sealant cannot be predicted from these results; nor can it be said whether the Furan would be adversely affected by the higher temperatures (up to 170 C (340 F)) found in the cooking zone of a digester. In actual digester service, the Furan has been reapplied yearly, and no coating disbondment has occurred with Furan sealed thermal spray coatings since sealed coatings were first used in 1982 [23].

Applicability of Results to Digesters:

Under controlled laboratory conditions, SCC occurred readily in the control specimen when exposed to a simulated impregnation zone liquor. The ranking of digester repair and crack prevention methods produced by this test is consistent with previously reported laboratory data [3] and, in general, these results are also consistent with experience in digesters [1,20]. Despite the fact that crack initiation and propagation in the CPTW specimens was accelerated by the use of a controlled, worst-case cracking potential, crack propagation rates measured in these specimens of between 1 and 24 mm/y (0.03 and 1 in/yr) are similar to maximum crack propagation rates estimated for digesters in service [1]. Similarly, although the CPTW specimens were highly constrained, residual tensile stresses measured in the specimens did not differ significantly from those found in digester welds. Values as high as 400 MPa (58 ksi) have been measured in non-stress relieved welds in a continuous digester in which severe

Inconel 82 and 309L SS Weld Overlay: The weld overlays were deposited as a series of beads over the carbon steel weld, much as would be done in a digester. Both sides of each specimen were overlaid. One half of each face of the specimens was subsequently shotpeened (both the overlay and base metal), while the other half was left unpeened as a control.

In the specimen overlaid with Inconel 82, a substantial, circumferential stress corrosion crack was found on one face of the specimen, in the notch at the interface between the outer edge of the overlay and the base plate (Figures 16,17). The depth of this crack was about 6 mm (0.25 in) at the point where the specimen was sectioned. A similar, shallower crack (0.5 mm (0.02 in)) was also found on the opposite face in the outer overlay/base metal interface. Both cracks extended around the major portion of the unpeened section of the specimen. Some short transverse cracks were observed in the base plate immediately adjacent to the inner edge of the Inconel 82 overlay on both faces. No cracks were observed in the interfacial areas which had been shotpeened. Significantly, residual tensile stresses were highest close to the outer edge of the overlay with the deepest circumferential crack.

No cracks were found on the interface between the base plate and the outer edge of the 309 SS overlay, but some short, transverse cracks were found in the base plate adjacent to the inside edge of the overlay (Figures 18,19). As with the specimen overlaid with Inconel 82, no cracks were found in the shotpeened sections of the specimen. Consistent with the lack of SCC at the outer interface between the base plate and the overlay band, low tensile or compressive stresses were found in the base plate close to the overlay. The differences in magnitude and distribution of residual stress between the Inconel and 309 SS weld overlay specimens is most likely attributable to differences in specimen preparation. It is expected that weld overlays of either metallurgy would be equally at risk in a digester.

These results strongly suggest that, if sufficient residual tensile stress is present at the interface between the base plate and weld overlay bands (and all other necessary conditions for SCC are satisfied), SCC is likely to occur at the overlay/carbon steel interface in digesters. Recent tests on large plates simulating weld overlays in digesters have shown that residual tensile stresses near the yield strength of the material may exist at the base plate/weld overlay interface [21]. Stresses of this magnitude were more than sufficient to initiate SCC in these tests. Since cracking has been reported in some digesters at the interface between weld overlays and the underlying base plate [20], it may be prudent for digester operators to schedule periodic inspections for cracking at the overlay/base metal interface in any digester which has had overlays applied over the shell welds.

Sealed Thermal Spray Coatings: One face of this specimen was plasma sprayed with Metco 465 powder, while the other was flame sprayed with Metco 444 powder. A modified Furan sealant was then brushed over the entire surface of the specimen. The sealant was still largely intact at the conclusion of the test period. As a consequence, the specimen was not inspected for SCC. A visual examination revealed no differences between the two faces at the conclusion of the test, indicating that the Furan coating had adhered equally well to both thermal spray coatings (Figure 20). The Furan had darkened considerably over the exposure period, and had also suffered some disbondment and slight mechanical damage over rough spots on the surface and edges of the specimen. It

Stress measurements showed that residual compressive stresses were present on the surface of the base metal adjacent to the outside of the weld. In general, residual tensile stresses present in the temper-bead welded specimen were much lower than in the control specimen. However, residual tensile stresses similar in magnitude to the control specimen were measured on the base plate near the inside edge of the weld, where the cracking occurred. The beneficial effect of temper-bead welding is not generally considered to be a reduction in residual tensile stress, but rather a modification of the weld and HAZ microstructures to softer, less crack-susceptible forms. It is not clear whether the observed reduction in residual tensile stress is an artifact of the specimen design, or whether it could also be achieved in a digester weld that was prepared using temper-bead welding. Some of the benefit of temper-bead welding in this case likely resulted from the reduction of residual tensile stresses in the specimen.

Shotpeening: One half of each face of this specimen was shotpeened, while the other half was masked off as a control. Residual compressive stresses as high as -291 MPa (-42 ksi) were measured at the peened surface, while residual tensile stresses at the yield strength of the material were recorded for the unpeened weld metal and base metal adjacent to the weld (similar to the control specimen). No SCC originated in the shotpeened sections of the specimen (Figures 14,15). In Figure 15, a major circumferential crack can be seen penetrating a short distance into the shotpeened area. It is likely that this crack propagated underneath the shotpeened section of the specimen (in an area of high residual tensile stresses) before breaking through to the surface. This situation would not likely arise in a shotpeened digester, where the entire weld area is shotpeened.

Although shotpeening has been shown to be an effective means of preventing SCC in these laboratory tests, re-cracking has been observed in some shotpeened digesters, even after multiple applications [1]. Note that a significant difference between these tests and digesters is that little or no general corrosion occurred on the CPTW specimens over the time period of the test. This is contrary to experience in digesters, where significant pitting and general corrosion is often observed. The long term durability of the compressive layer introduced by shotpeening in digesters is therefore in doubt because this layer is very thin and could quickly be removed by excessive corrosion of the digester shell (eg., particularly during acid cleaning). Shotpeening is not normally recommended as a method for preventing SCC when corrosion is also expected to occur.

Shotpeening was also effective in preventing SCC in carbon steel at the interface between the Inconel 82 and 309 stainless steel weld overlays and the underlying plate. The possibility of such cracking in digesters has recently become a concern [20], and shotpeening may provide a means of reducing the susceptibility to cracking in these areas for mills which have extensively overlaid welds in their digesters. Note that the same disadvantages apply to shotpeening the overlay/base metal interface as shotpeening the entire weld and that the effectiveness of shotpeening in such areas is also dependent on the shot being able to completely penetrate to the root of the notch. Thus, a fairly smooth weld profile at the overlay/base metal interface would be required prior to shotpeening.

Prior to being stress relieved, the residual tensile stresses in this specimen had been comparable in magnitude to the control specimen. This result reinforces the evidence that stress relief of a digester is beneficial for crack prevention.

Although stress relief was completely effective in preventing SCC in this specimen, cracking has been reported in digesters which have been partially or wholly stress relieved [1]. Similarly, repair welds which were locally stress relieved have been reported to have re-cracked in some cases [1]. In some instances, the cracking may have been mis-identified as SCC [15]. In others, the residual tensile stress left after stress relief, combined with operating stresses in the digester, may have been large enough to cause SCC. Typical tensile stress levels in welds of stress relieved digesters are not known, but tensile stresses as high as 141 MPa (20 ksi) have been measured in a repaired digester weld after local stress relief [17]. In combination with operating stresses imposed on the vessel, a residual tensile stress of this magnitude may be sufficient to cause SCC. Note also that low-cycle periodic stressing, such as might be experienced by operating digesters, is known to significantly lower the minimum tensile stress required to initiate SCC in some carbon steels [18].

<u>E6010 Capping Pass</u>: Very little cracking was found on the first face of the specimen to be welded. More extensive cracking was found on the second face, but apparent crack depths were shallower than for the cracks found on the control specimen and the crack orientation was predominantly transverse to the weld (Figures 10,11). Residual tensile stresses in the weld and in the base plate adjacent to the weld were similar in magnitude to those in the control specimen. Previous laboratory testing [3] has also shown that E6010 weldments are more resistant to SCC than E7018 weldments, most likely as a result of the softer, more refined microstructure and lower yield strength of E6010 weld metal.

Note that E6010 welds are more susceptible to hydrogen-induced cold cracking than E7018 welds due to a higher hydrogen content in the electrode coating and thus require more care when welding [14]. Cold cracks and other weld defects can act as stress raisers and initiation sites for SCC, so it may not be practical to use E6010 capping passes as a means of reducing the severity of SCC in digesters. Although no carbon steel weld or base metal is immune to SCC, low hydrogen electrodes which possess the desireable characteristics of the E6010 electrode for stress corrosion resistance, namely, a soft, fine-grained microstructure and lower tensile strength, could be considered for capping passes in digester construction and repair.

<u>Temper-Bead Welding</u>: Temper-bead welding is a procedure designed for weld repair in locations where post-weld heat treatment is difficult [19]. The hardness is minimized in the weld and in the heat-affected zones by overlapping weld beads such that each bead is refined by a subsequent pass. SCC (and other types of cracking) is expected to be less severe when such welding procedures are employed. In this specimen, cracking was only observed on the last face of the specimen to be welded, in the weld pass closest to the inside circumference of the specimen (Figures 12,13). No cracking was found on the first face of the specimen. The cracks were all very short and oriented transverse to the weld. The maximum apparent depth of penetration of any crack was less than 0.050" (1.3 mm), although actual crack depths for two cracks were found to be much larger.

and the maximum apparent crack depth are compared for each specimen. Due to the limited number of actual crack depth measurements made on the specimens (Table I), these values have not been used for purposes of comparison between specimens.

Extremely severe cracking occurred in the weld of the specimen made with the worst-case welding procedure. Moderate SCC occurred in the control specimen, and less severe cracking was observed in the specimen capped with a 6010 welding pass, and in the temper-bead welded specimen. SCC was also observed in carbon steel at interfaces between the base plate and both weld overlays. No cracking occurred in the stress relieved specimen or in the specimen protected by sealed thermal spray coatings. Minor degradation of the Furan sealant on the thermal sprayed specimen was observed, but the sealed coatings prevented cracking over the test period. No cracking occurred on areas of specimens which were shotpeened. A detailed discussion follows for each specimen.

Standard Weld Procedure: This specimen is a simulation of a typical weld in a kraft continuous digester, and is the control against which all of the other specimens were judged. SCC observed in this specimen closely resembled cracking found in digesters (Figure 3). Both faces of the specimen were cracked in the weld metal and in the weld HAZ. Most cracks were oriented parallel to the weld, but short cracks were also found transverse to the weld (Figures 4,5). The deepest cracks (0.215" or 5.5 mm) were found in the weld HAZ. Residual stress measurements showed near-yield strength tensile stresses in the weld metal and in the base metal close to the fusion lines of the weld. The largest stresses were oriented transverse to the weld, consistent with the orientation of most cracks.

No Preheat and Ambient Interpass Welding Temperatures: The welding procedure used for this specimen represents the worst-case procedure for a field or shop weld. As expected, residual tensile stresses were extremely high in this specimen. A single deep stress corrosion crack ran around nearly 3/4 of the weld circumference on the "B" side of the specimen (Figures 6,7). Clearly visible to the naked eye, the depth of the crack varied between 6.4 mm (0.25") and 12.7 mm (0.50") for much of its length. Possibly as a result of the rapid severe cracking which occurred on this side of the specimen, very little cracking was observed on the opposite face.

The severity of cracking in this specimen is a strong indication of what can occur in a digester when established welding procedures are not followed. The need for proper temperature control before, during, and after welding to minimize residual tensile stresses in the weld and to produce a lower hardness weld microstructure is well known [13,14]. In addition to increasing susceptibility to SCC, poor welding procedures can often lead to hydrogen-induced cold cracking [14]. Such cracks are also found in digesters [15], and are almost always due to the use of an improper weld procedure (particularly a lack of temperature control, or the presence of moisture during welding).

Stress Relief: The stress relief procedure for this sample was consistent with that specified for digesters by the ASME Boiler and Pressure Vessel Code [16]. No stress corrosion cracks were found on either side of this specimen (Figures 8,9). Stress measurements showed that minimal residual tensile stresses were left in the weld and in the base metal adjacent to the weld after stress relief.

severity of cracking which occurred in these specimens over such a short time period confirmed that a specimen of this design would be suitable for SCC testing in liquor compositions more typical of those found in kraft continuous digesters. Since SCC was more severe in the larger specimen, the design chosen for further testing consisted of a 254 mm (10") diameter, full-thickness disk cut from a 38 mm (1.5") thick plate of digester steel (Figure 2).

Ten specimens (nine for testing and one spare) were prepared. Crack prevention techniques which were evaluated included stress relieving, shotpeening, sealed thermal sprays, and the application of Inconel 82 and 309L stainless steel weld overlays. In addition, different weld procedures were evaluated for their tendency to enhance or reduce the severity of SCC, compared to a typical digester welding procedure. These were a specimen welded with no pre-heat and at ambient interpass temperatures (considered the worst-case welding procedure), a temper-bead welding procedure and the use of a 6010 capping pass. Further details of the specimen design and welding procedures are given in the experimental section.

Residual tensile stresses in the CPTW specimens were measured prior to exposure to the liquor by a blind hole drilling method [12]. Details of the measurement technique are given in the experimental section. In general, residual tensile stresses near yield-strength (>340 MPa or 50 ksi) were measured in the weld metal or in the weld heat-affected zones (HAZ) of the control specimen, in the worst-case welded specimen, and in the specimen welded with a 6010 capping pass. Tensile stresses as high as 250 MPa (36 ksi) were measured in the temper-bead welded specimen and adjacent to the outside edge of the Inconel weld overlay specimen. Residual tensile stresses of less than 1 MPa (0.1 ksi) were measured in the stress-relieved specimen.

The test method was made as realistic as possible by exposing the specimens at 115 C (234 F) to the simulated impregnation zone liquor (40 g/L NaOH + 20 g/L Na_2S + 20 g/L Na_2CO_3) used by Paprican for previous stress corrosion testing [3]. The test duration was 150 days. Crack initiation and propagation rates in the specimens were maximized by controlling the electrochemical potential of the specimens to the worst-case cracking potential previously determined for this liquor [3].

Evaluation of Crack Prevention Methods:

The specimens were evaluated for SCC by mapping the extent of cracking found during a wet fluorescent magnetic particle (WFMP) inspection. Since the specimens were completely welded on one face ("A" side) before welding the other ("B" side), higher residual tensile stresses and more severe SCC were found in the last face to be welded. Apparent crack depths were determined using a resistance-type crack depth gauge. Depth gauge measurements were subsequently compared to actual crack depths by sectioning each specimen at a randomly chosen location on each specimen and measuring the depth of the cracks which were revealed (Table I). The crack depth indicator was found to be accurate to within ±20% for long, deep cracks, but was much less accurate for short cracks. In all cases, actual crack depths were compared to the recorded depth gauge measurements nearest to the cross-section.

A summary of results for all specimens is given in Table II. Visual inspection results, the average apparent crack depth measured by the depth gauge, the test specimen should be such that the application of the technique closely resembles actual application in the field. Similarly, it is preferable that the specimen (ie., size, welding procedures) and test environment (ie., liquor composition, temperature) simulate digester conditions as realistically as possible. Finally, it is essential that SCC of the kind observed in digesters be induced in a control specimen.

Test methods which had been previously been used by Paprican to investigate digester cracking (ie., slow strain rate and fracture mechanics techniques) were well suited for investigation of the mechanism of cracking in digesters and for an evaluation of the effectiveness of anodic protection against SCC. However, they could not be adapted to evaluate the effectiveness of applying barrier-type crack prevention methods such as sealed thermal sprays and weld overlays, or of using different weld procedures. Test methods employed by Battelle to evaluate barrier-type digester repair methods were unsuccessful because SCC did not occur in their control specimens [2]. Thus, a novel specimen design was required to evaluate stress corrosion repair methods for digesters.

The test specimen chosen by Paprican was based upon the circular patch test weld (CPTW) specimen commonly used for welding procedure qualifications [7,8]. Specimens of this type usually consist of a section of full-thickness plate from which a central disk has been cut out and welded back in place. The severe residual tensile stresses left in the welds of such specimens ensure that any cracking related to the welding procedure (eg., cold cracking) will be detected before the weld procedure is employed in the field.

This particular specimen design offered several features which made it ideal for the evaluation of methods for the prevention of SCC in digesters. Specimens could be made from full-thickness digester plate and welded according to standard or modified digester welding practices. The constraints imposed on the specimen guaranteed the presence of high residual tensile stresses and thus, maximum susceptibility to SCC. In addition, the specimens could be made large enough to allow practical application of both weld overlays and sealed thermal spray coatings. Limited use has been made in the past of similar specimens for SCC testing [9,10].

Specimen Design and Test Method:

Two CPTW specimens were fabricated for initial testing. Both were full-thickness disks cut from a plate of 38 mm (1.5") thick digester steel. One specimen was made with an overall diameter of 305 mm (12"), while the other had an overall diameter of 152 mm (6"). The specimen design differed slightly from a normal CPTW specimen in that the central disk was not cut completely free of the specimen before welding. This avoided problems with alignment of the disk during welding and ensured an even stress distribution around the weld.

The susceptibility of these specimens to SCC was evaluated by exposing them to a solution containing 33% NaOH at 100 C (212 F), an environment known to produce severe SCC in carbon steels [11]. After an exposure of approximately 7 days at a controlled, worst-case cracking potential, severe cracking was induced in the welded portions of both specimens (Figure 1). The

II. INTRODUCTION

Following the failure of a kraft continuous digester in 1980 as a result of stress corrosion cracking (SCC), the Digester Cracking Research Committee (DCRC), jointly administered by TAPPI and the Institute of Paper Chemistry (IPC), was formed to support research into the causes and prevention of this industry-wide problem. Research for the DCRC was carried out at three laboratories: IPC, the Pulp and Paper Research Institute of Canada (Paprican) and Battelle Memorial Institute, Columbus Laboratories. This program was completed, and final reports issued by the laboratories, in late 1984 [1-3]. However, several outstanding issues remained at the conclusion of the program. In particular, one unfulfilled objective was the development of a laboratory test method capable of predicting the long-term performance of crack prevention measures used in digesters. A decision was therefore made by the DCRC steering committee to support the development of such a test method at Paprican.

Previous reports issued by Paprican to the DCRC have dealt with the effects of acid cleaning, liquor composition, temperature, digester metallurgy, and tensile stress on digester cracking [3-5]. The effectiveness of anodic protection for crack prevention was also evaluated [3,6]. Caustic SCC was identified as the mechanism for much of the cracking found in digesters. No evidence was found that cracking in digesters was related to acid cleaning [3,4]. However, it was found that severe general corrosion and pitting of the digester shell could occur during acid cleaning. The electrochemical potential of the digester, and the magnitude of residual tensile stresses present in the digester shell are the two variables which were determined to have the most influence on the severity of cracking in a digester [3]. Susceptibility to cracking was not significantly affected by changes in liquor composition or temperature within the ranges commonly found in the impregnation and cooking zones of a kraft continuous digester. Cracking occurred in all digester plate and weld metal compositions investigated [3], although experimental results indicated that the use of welding electrodes and procedures which left a soft, fine-grained weld microstructure could reduce the severity of cracking [3]. Anodic protection was determined to be an effective means of preventing SCC in digesters [3,6].

In this report, results of the supplementary research program are described. The principal aim of this research was to develop an accelerated test method for the evaluation of techniques used to prevent SCC in digesters. The suitability of the test method for this purpose was determined by evaluating nine different crack prevention methods and welding procedures for their relative ability to prevent stress corrosion cracking (SCC) under simulated digester operating conditions. The results were then compared to long-term experience in digesters.

III. RESULTS AND DISCUSSION

Methodology:

The primary requirement of a test method for the evaluation of crack prevention techniques is that it must predict, within a reasonably short time span, the long-term abilities of the techniques to prevent SCC. The design of

I. CONCLUSIONS

1) An accelerated laboratory SCC test method was developed for full-thickness weldments which tests the effectiveness of stress corrosion crack prevention methods used in digesters.

2) Results from experiments conducted in a simulated digester environment using this test method support the following conclusions on the prevention of SCC:

   (a) High residual tensile stresses and severe SCC can result when inadequate preheat, interpass and post-weld temperatures are maintained during construction or repair welding.

   (b) Stress relieving reduces residual tensile stresses in welds and can significantly reduce susceptibility to SCC.

   (c) In the presence of sufficient residual stress, severe SCC of carbon steel is possible at the interface between Inconel 82 and 309 SS weld overlays and the underlying base plate.

   (d) Shotpeening prevented initiation of SCC on weld surfaces and at the interface between weld overlays and the underlying carbon steel base plate. Note that shotpeening would not be effective in cases where corrosion or pitting occurred in addition to cracking.

   (e) Sealed plasma and flame spray coatings can prevent SCC of welds. Minor degradation of the Furan sealant was observed over the test period.

   (f) Temper-bead welding procedures can reduce the severity of SCC in welds as compared to conventional welding procedures.

   (g) Capping passes of E6010 were found to reduce the severity of SCC as compared to conventional E7018 welds.

RESUME

Une méthode d'essais accélérés en laboratoire a été développée pour évaluer l'efficacité des moyens de prévention de la corrosion sous tension utilisés dans les lessiveurs en continu kraft. La pertinence de cette méthode a été déterminée en évaluant neuf différents moyens de prévention de fissure et procédés de soudure sous des conditions simulées d'opérations d'un lessiveur. Les résultats ont été, par la suite, comparés à l'expérience de longue date dans les lessiveurs.

De très sévères fissurations dans le soudure et dans la zone environnante affectée par la chaleur sont apparues sur un spécimen préparé par le pire procédé de soudure. Une corrosion fissurante modérée est apparue sur l'échantillon de contrôle et une moins importante fissure a été observée sur un spécimen recouvert d'une passe de soudure 6010 et sur un autre au cordon de soudure trempé. Le même type de fissuration a aussi été observé sur les aciers au carbone aux interfaces entre la plaque de base et les deux enduits de soudure. Dans le cas de l'enduit Inconel, une fissure s'était propagée à plus de 6 mm (0.25") dans la plaque de base. Aucune fissuration n'est apparue sur un spécimen ayant subit un traitement de relaxation et sur un autre protégé par un recouvrement étanche pulvérisé à chaud. Sur ce dernier, une légère dégradation de la couche primaire Furan a été observée alors que les couches étanches ont empêché efficacement la fissuration au delà de la période d'essai. Aucune fissuration n'est apparue sur des specimens dont les surfaces ont été mises en compression par grenaillage.

Ces résultats, en général, correspondent bien avec l'expérience de longue date dans les lessiveurs. Par conséquent, cette méthode d'essais accélérés s'avère être un excellent moyen d'évaluer l'efficacité relative de différentes mesures de prévention de la corrosion sous tension que l'on prévoit utiliser dans les lessiveurs.

SUMMARY

An accelerated laboratory test method was developed to evaluate the effectiveness of techniques used to prevent stress corrosion cracking (SCC) in kraft continuous digesters. The suitability of the test method for this purpose was determined by evaluating nine different crack prevention methods and welding procedures under simulated digester operating conditions. The results were then compared to long-term experience in digesters.

Extremely severe weld and heat-affected zone SCC occurred in a specimen made with a worst-case welding procedure. Moderate SCC occurred in the control specimen, and less severe cracking was observed in a specimen capped with a 6010 welding pass, and in a temper-bead welded specimen. SCC was also observed in carbon steel at interfaces between the base plate and two different weld overlays. In the case of an Inconel overlay, a stress corrosion crack propagated more than 6 mm (0.25") into the base plate. No cracking occurred in a stress relieved specimen or in a specimen protected by sealed thermal spray coatings. Minor degradation of the Furan sealant on a thermal sprayed specimen was observed, but the sealed coatings effectively prevented cracking over the test period. No cracking occurred on areas of specimens which were shotpeened.

These results generally correlate well with longer-term experience in digesters. Therefore, this accelerated test method appears to be an excellent means of assessing the relative effectiveness of various stress corrosion prevention measures considered for use in digesters.

KEYWORDS

COATINGS, CONTINUOUS PROCESS, CORROSION, CORROSION TESTS, CRACKS, DIGESTERS, FRACTURE, KRAFT LIQUORS, PROTECTIVE COATINGS, SEALANTS, STEEL, STRESS CORROSION, STRESSES, TEST METHODS, WELDED JOINTS, WELDING